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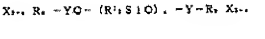
(54) AQUEOUS SILICONE EMULSION WHICH FORMS SILICONE ELASTOMER HAVING IMPROVED ADHESION TO SUBSTRATE

(57)Abstract:
PROBLEM TO BE SOLVED: To obtain an aqueus silicone emulsion having improved storage stability and adhesion by mixing water with a surfactant, a crosslinking agent, a tin condensation catalyst, a specified diorganosiloxane polymer, a specified amino-functional siloxane and an acid.
SOLUTION: A diorganosiloxane polymer represented by formula I [wherein (n) is 0-3; (z) is 200-15; X is hydroxyl or a hydrolyzable group; R is a 1-5C (un)substituted monovalent hydrocarbon group; R1s are each X or R, provided that at least 90% of them are Rs; Y is Si, -Si-(CH2)mSiR12-group,-Si-(CH2)m-SiR12- SiR12-(CH2)mSiR12-group and (m) is a positive integer] is mixed with water, a surfactant, a crosslinking agent, a tin condensation catalyst, an amino-functional siloxane prepared by reacting an amino-functional silane represented by formula II [wherein A is a hydrolyxable group; Z is O or NR2; R2 is H or a 1-15C (un)substituted monovalent hydrocarbon group; (p) and (q) are each 2-10; and (r) is 0-3] with a hydroxyl-terminated organosiloxane represented by formula III (wherein R4 is R; and (b) is 4-80) and an acid.



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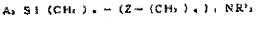

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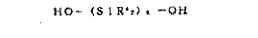

III

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CLAIMS

[Claim(s)]

[Claim 1]JIORUGANO siloxane polymer (I) which is a drainage system silicone emulsion which forms a silicone elastomer which has an improved adhesive property to a substrate when water is removed, and is shown with a silicone emulsion:(A) following formula containing the following

$X_{3-n}R_n$–YO–$(R^1{}_2SiO)_z$–Y–R_nX_{3-n} — here, n is 0, 1, 2, or 3, and z is an integer of 200–10,000, X is a hydronalium SHIKIRU group or a certain hydrolytic basis, and R individually, 1–15 carbon atoms were replaced, or it is an unsubstitutad univalent hydrocarbon group, R^1 is individually chosen from X basis and R group, however at least 90% of R^1 is R group, Y And an Si atom and –Si–$(CH_2)_m SiR^1{}_2$–basis, Or (B) water whose m it is a –Si–$(CH_2)_m SiR^1{}_2$–O–$SiR^1{}_2$–$(CH_2)_m SiR^1{}_2$–basis, and is as having defined R^1 here above, and is a positive integer;
(C) A surface-active agent;
(D) Arbitrary cross linking agents;
(E) A tin condensation catalyst;
(F) Amine functionality Silang (II) shown with a following formula
$A_3Si(CH_2)_p$–$(Z–(CH_2)_q)_r NR^3{}_2$ (here) A is a hydrolytic basis and Z is an oxygen atom or NR^2 (chosen out of a univalent hydrocarbon group by which a hydrogen atom and 1–15 carbon atoms were replaced individually, or R^2 is not replaced here), R^3 is chosen from a univalent hydrocarbon group which a hydrogen atom and 1–15 carbon atoms were replaced individually, or is not replaced, and p and q are the positive integers of 2–10, respectively — r — an integer of 0–3 — it is — with an included ingredient. Formula HO–$(SiR^4{}_2)_b$–OH (R^4 is chosen from a univalent hydrocarbon group which 1–15 carbon atoms ware replaced individually, or is not replaced here, and) b — a positive integer of 4–80 — it is — effective dose of amino functionality siloxanes; formed by making hydroxy end ORGANO siloxane (III) shown react, and (G) — arbitrary acid.

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DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[Field of the Invention]This invention relates to the drainage system silicone emulsion which forms the silicone elastomer which has an improved adhesive property to a substrate. when water is removed.
[0002]
[Description of the Prior Art]Document is teaching that a drainage system silicone emulsion is a dispersing element of underwater siloxane polymer. Bridge construction of siloxane polymer has the more common bridge construction before evaporation of water. although the front stirrup of evaporation of water may happen behind. If water is evaporated, a silicone emulsion will form a tunic, sealant, and the silicone elastomer of the form of a caulking agent.
[0003]Generally, the silicone elastomer formed from the aquosity silicone emulsion has the outstanding weatherability, moderate high temperature oxidation stability, and good low-temperature characteristic. Reinforcement of an elastomer is required in order for these silicone elastomers to have a remarkable mechanical property. This reinforcement is attained by use of various fillers, for example, a colloidal silica, precipitated silica, or fumed silica, or use of the silica formed on that spot from the silicon content compound precursor.
[0004]On account of those characteristic profiles, the silicone elastomer has found out the use as an additive agent widely used for building sealant, a tunic, a caulking agent. and a building. It is added depending on a specific use, for example, a thickener, a rheology denaturing agent, a dispersing agent, paints, a flatting, a defoaming agent, an adhesion promoter, and **** stabilizer, and deals in other ingredients.
[0005]I hear that they lack the good adhesive property to a substrate, and one fault of the silicone elastomer obtained from a drainage system silicone emulsion has them. Considerable efforts have been paid in order to find out the method of improving the adhesive property of such a silicone elastomer. This advanced technology is widely represented by GB-A 2152521, JP-A 58/69250, and US-A 3817849, 4228054, 4412035, 4496687, 4535109, 4710405, and 4877828.
[0006]Although the described method brought the better adhesive property to the silicone elastomer, a problem still exists. When net amino functionality Silang is added to a silicone emulsion, it is making an emulsion unstable, making storage life scarce, and bringing about condensation or coagulation. In addition, since an adhesion promoter will be deactivated if it hydrolyzes, from two to six weeks, any improvements of the adhesive property acquired by addition of net amino functionality Silang continue, and are not maintained in many periods.
[0007]
[Problem(s) to be Solved by the Invention]The purpose of this invention is to prepare the drainage system silicone emulsion which forms the silicone elastomer which gives the adhesive property improved when water is removed to a substrate. Other purposes are to prepare the emulsion which forms a silicone elastomer, when it has the storage life improved when it is in the state of an emulsion and water is removed. The last purpose is to continua at a long period of time and to maintain storage life and an adhesiva improvement.
[0008]
[Means for Solving the Problem]The purpose of this invention is attained by preparing a drainage system silicone emulsion which forms an elastomer which has an improved adhesive property to a

substrate, when water is removed. This emulsion JIORUGANO siloxane polymer, water, a surface-active agent, Output formed by mixing arbitrary cross linking agents, a tin condensation catalyst, amino functionality Silang, a hydroxy end ORGANO siloxane, and an effective dose of amino functionality siloxanaa formed by making arbitrary acid react is included.
[0009]This invention is a drainaga system silicone emulsion, : (A) which is a thing including output formed by mixing the following — JIORUGANO siloxane polymer $X_{3-n}R_nSiO(R^1_2SiO)_z-Y-R_nX_{3-n}$ shown by general formula (I) of the following — here, n is 0, 1, 2, or 3, and z is an integer of 200-10,000, X is a hydronalium SHIKIRU group or a hydrolytic basis, and R is [1-15 carbon atoms were replaced individually, or] an unsubstituted univalent hydrocarbon group, R^1 is individually chosen from X basis and R group, however at least 90% of R^1 is R group, Y And an Si atom and $-Si-(CH_2)_mSiR^1_2$-basis, Or (B) water whose m it is a $-Si-(CH_2)_mSiR^1_2-O-SiR^1_2-(CH_2)_mSiR^1_2$-basis, and is as having defined R^1 here above, and is a positive integer;
(C) Surface-active agent;
(D) Arbitrary cross linking agents;
(E) Tin condensation catalyst;
(F) Amino functionality Silang (II) shown with a following formula
$A_3Si(CH_2)_p-(Z-(CH_2)_q)_rNR^3_2$ (here) A is a hydrolytic basis and Z is an oxygen atom or NR^2 (R^2 is chosen from a univalent hydrocarbon group which a hydrogen atom and 1-15 carbon atoms were replaced individually, or is not replaced here), R^3 is chosen from a univalent hydrocarbon group which a hydrogen atom and 1-15 carbon atoms were replaced individually, or is not replaced, and p and q are the positive integers of 2-10, respectively — r — an integer of 0-3 — it is — with an included ingredient. Formula $HO-(SiR^4_2)_b-OH$ (R^4 is chosen from a univalent hydrocarbon group which 1-15 carbon atoms were replaced individually, or is not replaced here, and) b — a positive integer of 4-80 — it is — effective dose of amino functionality siloxanes; formed by making hydroxy end ORGANO siloxane (III) shown react, and (G) — arbitrary acid.
[0010]An ingredient (A) is JIORUGANO siloxane polymer. A term "JIORUGANO siloxane polymer" used here means a siloxane constituent which has a mixture containing a single kind of siloxane polymer, or its mixture. This JIORUGANO siloxane polymer may be a homopolymer, a copolymer, or a terpolymer. This term includes a line of various molecules, for example, a long-chain line, and a molecule of branching, or a short chain, and a molecule of branching. although it is not important for this invention — viscosity of said organosiloxane polymer — the range of 5000 - 500,000 mPa-s — it is the range of 10,000 - 100,000 mPa-s preferably. However, if viscosity is adjusted using a solvent, polymer of a higher molecular weight, a polymer blend, etc. can be used.
[0011]"JIORUGANO siloxane polymer" used here is well-known at a person skilled in the art. They are described by the above-mantioned formula (I), and it is commercially available or they are made by a publicly known method.
[0012]The substituent X is a basis combined with 1 or a silicon atom beyond it, and is a hydroxyl group or a hydrolytic basis. A hydrolytic basis contains all the bases combined with silicon hydrolyzed with water at a room temperature. As an example of the suitable hydrolytic basis X, a hydrogen atom; halogen atom, for example, chlorine, a basis (here — T — a certain hydrocarbon group or a halogenated hydrocarbon group.) shown by bromine, fluoride. and iodine; type-OT For example, methyl, ethyl, isopropyl, octadecyl, allyl, hexenyl, Cyclohexyl, phenyl, benzyl, beta-phenyl ether, 2-chloroethyl, they are chlorophenyl, 3,3,3-trifluoropropyl, and bromocyclohexyl —; — a hydrocarbon ether group of some kind, For example, 2-methoxy ethyl, 2-ethoxy isopropyl, 2-butoxy isobutyl, p-methoxyphenyl and $-(CH_2CH_2O)_2CH_3$; An acyl group of some kind, For example, acetyl, propionyl, benzoyl, cyclohexenyl, AKURIRIRU, Methacrylyl, stearyl, naphthoxy one, trifluoroacetyl, chlorobenzoyl, and bromopropionyl; An acyloxy group of some kind, For example, acetoxy, benzoyloxy, pro PIONOKISHI, and acryloxy; and a certain amino group, for example, NH_2, dimethylamino, diethylamino, ethyl methylamino, diphenylamino, There are methylphenylamino and dicyclohexylamino. X is a formula. - A certain aminoxy shown by ONT_2 or -ONT' (here) T is as having given the definition above and T' is a certain divalent hydrocarbon group, It is a certain; type at what both valences have

combined with carbon, for example, hexylene, pentylene, or octylene. - ON=CT_2 or -ON=CT' (here) as having defined T and T' above — It is — A certain kato KISHIMO group; type shown - N (T) — $CONT'_2$ (here) T is as having given the definition above, T'' A hydrogen atom. or either of the T bases — it is — it may also be a carboxylic amide group shown by carbamata group; or formula-NTC=O (T'') (it is as having defined T and T'' here above) shown by ureido group; type-OOCNTT'' (it is as having defined T and T'' here above) shown. X may also be a phosphate group or a phosphate ester group shown by formula-OSO_2(OT) (it is as having defined T here above); cyano group; isocyanate group;. or formula-$OPO(OT)_2$ (it is as having defined T here above) again.

[0013]As X, hydroxyl and an alkoxy group are preferred. As an example of an alkoxy group, there are methoxy and ethoxy ** propoxy, isobutoxy, pentoxy, HEKISOKISHI, and 2-ethyl HEKISOKISHI A basis much more desirable as X is hydroxyl.

[0014]The substituent R and R^1 express a univalent hydrocarbon group combined with a silicon atom with 1-15 carbon atoms which are not replaced or replaced, respectively. Although this may be the same or it may not be the same, at least 90% of R^1 groups are univalent hydrocarbon groups with 1-15 carbon atoms which are not replaced. As an example of a suitable univalent hydrocarbon group of R and R^1 which is not replaced or replaced, An alkyl group, for example, methyl, ethyl, isopropyl, hexyl, octadecyl, and myricyl; An alkenyl group, For example, vinyl, allyl, and hexenyl; An ARUKINARU (alkynel) group, For example, propergyl; Alicyclic group, for example, cyclopentyl, cyclohexyl, and a cyclohexenyl; aromatic hydrocarbon group, For example, phenyl, tolyl, xylyl, xenyl, naphthyl, and an anthra sill; An aralkyl hydrocarbon group, For example, benzyl, beta-phenylethyl, beta-phenylpropyl, and gamma-tolyl propyl; A halogenated hydrocarbon group or a halocarbon group. For example, there are 3,3,3-trifluoropropyl, the trideceafluoro 1,1,2,2, - tetrahydro octyl-1-methyl, and perfluoro-alkyl. In addition, R^1 is X.

[0015]R and R^1 are a methyl group or a methyl group, and a phenyl group preferably. R and R^1 is a methyl group much more preferably.

[0016]The substituent Y is single silicon atom (Si) or formula-Si-$(CH_2)_m$$SiR^1{}_2$- or -Si-$(CH_2)_m$$SiR^1{}_2$-O-$SiR^1{}_2$$(CH_2)_m$$SiR^1{}_2$- (here) R^1 is as having given the definition above — m — a positive integer — it is — a basis containing a methylene spacer group combined with silicon shown is expressed. Y is a silicon atom more preferably for this invention.

[0017]As long as it averages per molecule and at least 1.3 hydroxyls, hydrolytic bases, or these mixtures exist, said JIORUGANO siloxane polymer may exist as a mixture of different JIORUGANO siloxane polymer. Preferably, average per one molecule of JIORUGANO siloxane polymer, and at least 1.8 hydroxyls, hydrolytic bases, or these mixtures exist, and most preferably, it averages per one molecule of polymer, and at least two hydroxyls, hydrolytic bases. or these mixtures exist. JIORUGANO siloxane polymer which exists as a mixture is prepared mixing two or more individual JIORUGANO siloxane polymer before emulsification, or by mixing an emulsion which emulsified them individually and subsequently these-emulsified them.

[0018]As an example of desirable JIORUGANO siloxane polymer, hydroxyl terminal PDMS polymer and ethylene — doria — there are RUKOKISHI alkyl and PDMS polymer and ethylene dialkoxy alkyl silyl end PDMS polymer, and, Here, as an example of a desirable alkoxy group, it is methyl as an example of methoxy and those with ethoxy **, and a desirable alkyl group. Hydroxyl terminal PDMS polymer is more desirable JIORUGANO siloxane polymer.

[0019]An ingredient (B) is water. A silicone emulsion of this invention is an o/w emulsion. JIORUGANO siloxane polymer constitutes a disperse phase here, and a water layer constitutes a continuous phase. This water usually exists by eight to 1000 weight section on the basis of JIORUGANO siloxane polymer of 100 copies. Preferably, water recognizes 15-50 weight-section existence on the basis of 100 copies of same polymer as the above. Water may be added to once or can also be added at the time of versatility of a wish of a combination process. For example, in a desirable method of using emulsification directly, a JIORUGANO siloxane and an ingredient of a wish of others are emulsified using water of two to 5 weight section on the basis of a small amount of water, for example, JIORUGANO siloxane polymer 100 weight section. Subsequently, an obtained emulsion is diluted with additional water to a polymer solid content of a wish.

[0020]An ingredient (C) is a surface-active agent. A term "surface-active agent" describes a surface active agent chosen from a cationic surfactant, an anionic surfaotant, an ampholytic surface active agent, nonionic surface active agents. and these mixtures, and these stabiliza a disperse phase of an emulsion. Although each of these surface-active agents will be added individually, it will be combined with a surface-active agent of other kinds, and it will be added, but it is publicly known that it is useful although an emulsion of JIORUGANO siloxane polymer is stabilized.

[0021]As an example of a suitable cationic surfactant, fatty amines and those derivatives, For example, dodecyl amine acetate, octadecyl amine acetate, and acetate of amine of beef tallow fatty acid; A homolog of aromatic amine which has an aliphatic series chain, For example, dodecylanilline (dodecylanalin); Aliphatic series amide derived from aliphatic diamine, For example, undecylimidazoline; Aliphatic series amide derived from 2 substitution amine, For example, oleylaminodiethylamine; A derivative; quaternary ammonium compound which is ethylenediamine. For example, chloridation beef tallow trimethylammonium, dioctadecyl chloride dimethylanmmonium, Didodecyl chloride dimethylanmmonium and dihexadecyl chloride dimethylanmmonium; An amide derivative of amino alcohol. for example, beta-hydroxyethyl stearylamide; — amine salt [of long chain fatty acid]; — a quaternary ammonium base derived from fatty amine of 2 substitution diamine. For example, oleyl benzylamino ethylene diethylamine hydrochloride; A quaternary ammonium base of benzimidazoline, for example, methylheptadecylbenzimidezole bromate; pyridinium, a basic compound of the derivative, For example, cetyl pyridinium chloride; A sulfonium compound, for example, a quaternary ammonium compound of an octadecyl sulfonium methyl sulfate; betaine, For example, a betaine compound of diethylamino acetic acid or octadecyl chloromethyl ether; there are urathane [of ethylenediamine], for example, stearic acid, and condensation product; polyethylenediamine [of diethylenetriamine]; and polypropanol polyethylenediamine.

[0022]As an example of a suitable anionic surfactant, sulfonic acid which is indicated to US-A 3294725, and those salt derivatives exist. As an example of these anionic surfactants, sulfonation glyceral ester of alkaline metal sulfo RISHINETO (sulforicinate); fatty acid, For example, sulfonation monoglyceride of coconut oil acid; Sulfonation univalent alcohol ester, Sodium [for example,] oleylisethionate; Amide of aminosulfonic acid, for example, the oleyl MECHIRUTA reverse side — sodium salt [of ide]; — sulfonation output of fatty acid nitril. For example, palmitonitrile sulfonate; Sulfonation aromatic hydrocarbon. For example, condensation product; octahydro anthracene sulfone sodium which it is with the sodium; naphthalene sulfonic acid which is alpha-naphthalene mono-sulfone sodium and dibutyldodecylbenzenesulfonic acid (DBSA), and formaldehyde; Alkaline metal alkyl sulfate, For example, sodium lauryl sulfate; Eight carbon atom, Or ether sulfate which has an alkyl group beyond it; Dialkyl sulfonate in which alkyl aryl sulfonate; which has eight carbon atoms or an alkyl group beyond it 1 or more, and each alkyl group are more than eight carbon atome or it. For example, there is sulfosuccinic acid dioctyl.

[0023]As an example of a suitable ampholytic surface active agent, there are leoithin, glycinate, betainas, Sour Taine (sultaine), and alkylamino propionate. As these examples, coco-amphglyoinate (coco-amphglycinate), Coco-amphocarboxy-glycinate (coco-amphocarboxy-glyoinates), There are coco-amido propyl betaine, a lauryl betaine, ***** hydroxy-Sour Taine, lauryl SURUTEIN, and coco-amphodipropionate (coco-amphodipropionate).

[0024]As an example of a useful nonionic surface active agent, polyoxyalkylene alkyl ether, There are block copolymer of polyoxyalkylene sorbitan ester, polyoxyalkylene ester, polyoxyalkylene alkylphenyl ether, ethoxylation amide, an ethoxylation silioxane and propylene oxide, or ethyleneoxide, etc.

[0025]An anionic surfactant and a nonionic surface active agent are preferred. A nonionic surface active agent, for example, polyoxyalkylene alkyl ether, For example, a mixture of Tergitol(trademark) TMN-6, Tergitol(trademark) TMN-10, Triton(trademark)X-100 and a mixture of an anionic surfactant, for example, sulfosuccinic acid dioctyl, and a nonionic surface active agent is more preferred.

[0026]Generally, quantity of a surfaco-active agent used is a quantity which stabilizes a disperse phase of a silicone emulsion. 0.1 to 10 weight section is enough on the basis of JIORUGANO siloxane polymer 100 weight section. Preferably, quantity of a surface-active agent is 0.5 to 5 weight section on the basis of JIORUGANO siloxane polymer 100 weight section. Quantity of a surface-active agent is 1.5 to 5 weight section on the basis of JIORUGANO siloxane polymer 100 weight section more preferably.

[0027]An ingredient (D) is a cross linking agent. This ingredient is arbitrary depending on

JIORUGANO siloxane polymer. When this JIORUGANO siloxane polymer has a basis of many condensation nature or hydrolysis nature from an average of 1.3 per molecule, a cross linking agent for constructing a bridge in this JIORUGANO siloxane polymer is not required. A basis of condensation nature on a different JIORUGANO siloxane polymer molecule or hydrolysis nature forms a rubber particle which constructed the bridge in response to mutual, and it will dry, if water is ramoved from said emulsion, and this forms a silicone elastomer. Probably, it will be desirable to use it, though a cross linking agent is not required.

[0028]When said JIORUGANO siloxane polymer does not have a basis of many condensation nature or hydrolysis nature from an average of 1.3 per molecule, in order to form a rubber particle which will dry if water is removed from said emulsion, and forms a silicone elastomer, a cross linking agent must be added.

[0029]Typically, said cross linking agent is an organic silicon compound or resin containing a hydrolytic basis or hydroxyl which was averaged and was combined with many silicon atoms from two per molecule. A hydrolytic basis useful as a cross linking agent is the same as having described the substituent X of JIORUGANO siloxane polymer previously.

[0030]Said cross linking agent may also combine a basis of other kinds with silicon depending on a basis combined with JIORUGANO siloxane polymer, as long as it has these bases that combined preferably with at least three silicon a hydrolytic basis combined with much silicon from an average of two pieces, or hydroxyl. Molecular structure of this cross linking agent is the Silang structure or siloxane structure. A siloxane type cross linking agent may be a straight chain, branched chain, or annular. A hydrocarbon group which univalent [which indicated a hydrogen atom and R] was replaced as an exampla of a basis baaides hydrolytic Motomochi who has combined with a silicon atom of said cross linking agent, and is not replaced is included.

[0031]In short, any publicly known cross linking agents which construct a bridge in a drainage system silicone emulsion under existence of a tin condensation catalyst can use them for this invention, and as the example, There are colloidal silica, alkali silicate, alkali siliconate, alkoxysilane, an ARUKENOKI gardenia fruit run, an OKISHIMO silane, silazanes, a friend NOKISHI silane, silicone resin, and a silicon hydride. As an example of a special cross linking agent, a low-molecular-weight organicity silicon hydride, for example, a polymethyl hydrogen siloxane, A low-molecular-weight copolymer which has a methyl hydrogen siloxy group and a dimethyl siloxy group, $-OSi(Et)_2-$, ethylpolysilicate, and

$(OSiMe_2H_2SiOMe)_4Si$, $(OSi-ON=CR'_2)_4Si$, methyl trimetoxysilane, MECHIRUTORI propenoxysilane, and

tetraethyl orthosilicate, isobutyl trimethoxysilane, Ethyl triacetoxysilane, methyltriacetoxysilane, vinyltriacetoxysilane, There is vinyl trio KISHIMO silane and $Me_3SiO(Me_2SiO)_3(MeON(Et)_2)SiO)_2SiMe_2$, in each case, Me is methyl and Et is ethyl.

[0032]A desirable cross linking agent is Silang which has at least three alkoxy groups. A time of each alkoxy group having a carbon atom up to eight pieces is more preferred. A time of each alkoxy group having three carbon atoms is the most preferred, for example, it is a time of being methyl trimatoxysilane, isobutyl trimethoxysilane, methyl triethoxysilane, MECHIRUTORI propenoxysilane, and chloropropyl trimethoxysilane.

[0033]Usually, a cross linking agent is added by 0.1 to 50 weight section on the basis of JIORUGANO siloxane polymer 100 weight section, when used by this invention. It is preferably added by one to 10 weight section per JIORUGANO siloxane polymer 100 weight section, and is added by two to 5 weight section per JIORUGANO siloxane polymer 100 weight section still more preferably.

[0034]A cross linking agent is added as a single kind or a mixture of a thing of a different kind beyond 2 or it. This cross linking agent can be added before emulsification or to the back. However, it is preferred to add from an ease of combination, before emulsifying a cross linking agent.

[0035]An ingredient (E) is a tin condensation catalyst which is between this polymer when JIORUGANO siloxane polymer has sufficient hydrolytic basis, or is useful to promote crosslinking reaction between JIORUGANO siloxane polymer and a cross linking agent more typically. This catalyst is an organic tin compound of the valence+4, for example, a 2nd tin (IV) compound, and an organic tin compound of tin (IV) and the valence+2, for example, a mixture with a 1st tin (II) compound. A 2nd tin (IV) compound is preferred. As an example of the 1st tin compound, there are the 1st tin of oleic acid of carboxylic acid organicity, for example, tha 1st tin, the 1st tin of octanoic acid, and the 1st tin of neo decanoic acid. as the example of the 2nd tin compound — tin

SUTANNOKISAN (tin stannoxanes) — for example $[(C_4H_9)_2SnOI]_2O$ and $(C_4H_9)_2Sn(OI)-OSn(OH)(C_4H_9)_2$. Or there is formula $R'_2Sn(OCOR'')_2$ (R'' is individually chosen from a univalent alkyl group with 1–18 carbon atoms here, and R'' is R' or $-CH_2COR'$ individually (as R' was defined here above)). a tin catalyst — R'_2SnO, (a) $R'Sn(OCOR')_2$(b) $R'Sn(OCOR')_2$, the dicarboxylic anhydride; (c) carboxylic acid; (d) 2nd amine or, and N – acylation amino acid;R[(a) $]_nSn(SR')_{4-n}$ (here) a is 1, 2, or 3.; It may be a resultant with (f) carboxylic acid, alcohol, or alcohol amine. A tin (IV) compound is formula $R'_2DSn(OSnR'_2)_cD$ or R'_2SnD_2 (here). R' is as having given the definition above and D is dicarboxylic acid monoester. c is certain;M–$SnR'_2-(OSnR'_2)_x$–M (here) at a positive integer of 1–10. M may be an alkoxy group or tin, and other bases that can be coordinated, and x may be a compound expressed with certain; or $R'_2Sn(OSiR'_3)_2$ by a positive integer of 1–10.

[0036]Substituent R' is a univalent alkyl group with 1–18 carbon atoms. As an example of R', there are methyl, ethyl, propyl, hexyl, dodecyl, and octadecyl.

[0037]The substituent D is the dicarboxylic acid monoester (ethyl oxolate), for example, an ethyl oxo rate.

[0038]The substituents M are an alkoxy group or tin, and other bases that may be coordinated, for example, acetylacetonate.

[0039]The 2nd tin of carboxylic acid is a more desirable tin condensation catalyst, and dibutyltin diacetate is the most preferred.

[0040]In a silicone emulsion of this invention, it is sometimes desirable to reduce quantity of tin (IV). Cocatalyst with this publicly known, for example, Chu, H.K.;Crosse, R.P.; and Crossan, D.L.;J.Organomet.Chem., carboxylic acid that is indicated to 1992,425,9–17, or amine, And it is attained by using a salt of carboxylic acid or amine.

[0041]Generally, a tin condensation catalyst is added in quantity of 0.01 to 5 weight section on the basis of JIORUGANO siloxane polymer 100 weight section. Preferably, this catalyst is added in quantity of 0.05 per JIORUGANO siloxane polymer 100 weight section – the amount part of duplexs, and is added in quantity of 0.06 to 0.5 weight section per JIORUGANO siloxane polymer 100 weight section still more preferably.

[0042]A tin catalyst is a single kind or is added as a mixture of a different kind beyond 2 or it. The tin condensation catalyst can also add a front stirrup of emulsification behind. However, as for a tin catalyst, adding before emulsification is preferred because of an ease of combination.

[0043]An ingredient containing amino functionality Silang (II) an ingredient (F) is indicated to be by $A_3Si(CH_2)_p-(Z-(CH_2)_q)_rNR^3_2$. It is an amino functionality siloxane formed by making hydroxy end ORGANO siloxane (III) shown by formula $HO-(SiR^4_2)_b-OH$ react. Both said amino functionality Silang and a hydroxy end ORGANO siloxane are commercially prepared by a publicly known method available.

[0044]The substituent A is a hydrolytic basis. A hydrolytic basis also includes a basis of a gap to combine with silicon hydrolyzed with water at a room temperature. A suitable hydrolytic basis shown by A is a hydrolytic basis which indicated X.

[0045]The substituent A is an alkoxy group preferably and is an alkoxy group which has a carbon atom to 8 more preferably. As an example of an alkoxy group, there are methoxy and ethoxy ** propoxy, isobutoxy, pentoxy, HEKISOKISHI, and 2–ethyl HEKISOKISHI. An alkoxy group which has a carbon atom to 3 as A is the most preferred.

[0046]The substituent Z is an oxygen atom or NR^2 (R^2 is chosen from a univalent hydrocarbon group which a hydrogen atom and 1–15 carbon atoms were replaced individually, or is not replaced here). The substituent Z is NR^2 preferably. R^2 was replaced or an example of a univalent hydrocarbon group which is not replaced is the same as a case of the substituent R. R^2 is hydrogen preferably.

[0047]Inferior–letter p is a positive integer of 2–10, is 2–6 preferably, and is 3 most preferably.

[0048]Inferior–letter q is a positive integer of 2–10, is 2–6 preferably, and is 2 most preferably.

[0049]Inferior–letter r is an integer of 0–3, is 0–2 preferably, and is 0 or 1 most preferably.

[0050]Substituent R^3 is individually chosen from a univalent hydrocarbon group which 1–15 carbon atoms were replaced. or is not replaced. An example of these univalent hydrocarbon groups that is

not replaced and replaced is the same as what was given about the substituent R. Substituent R^3 may be a hydrogen atom. R^3 is a hydrogen atom preferably.

[0051]As an example of amino functionality Silang, $(CH_3O)_2Si-(CH_2)_3-NH_2$, $(C_2H_5O)_3Si-(CH_2)_3-NH_2$, $(CH_3O)_3Si-(CH_2)_3-NH-(CH_2)_3-NH_2$, $(CH_3O)_3Si-(CH_2)_3-O-(CH_2)_2-NH_2$, $(CH_3O)_3Si-(CH_2)_3-NH-(CH_2)_6-NH_2$, $(C_2H_5O)_3Si-(CH_2)_3-NH-(CH_2)_3-NH_2$, $(CH_3O)_3Si-(CH_2)_3-NH-(CH_2)_2-NH-(CH_2)_2-NH-(CH_2)_2-NH_2$. And (CH_3O) there is $_3Si-(CH_2)_3-NH-(CH_2)_2-N(CH_3)_2$.

[0052]It is made to mix with hydroxy end ORGANO siloxane (III) shown by formula $HO-(SiR^4{}_2)_b-OH$, and amino functionality Silang forms an amino functionality siloxane.

[0053]R^4 is individually chosen from a univalent hydrocarbon group which 1-15 carbon atoms were replaced, or is not replaced. An example of these univalent hydrocarbon groups that is not replaced and replaced is the same as what was given about the substituent R. Each R^4 is methyl preferably.

[0054]Inferior-letter b expresses a degree of polymerization in a hydroxy end ORGANO siloxane, and is 4-80. b is 4-50 preferably and b is 6-20 more preferably.

[0055]A hydroxy end ORGANO siloxane of a short chain is more preferred than a long-chain thing. That is because concentration of an effective amino functional group will be obtained with a comparatively small addition of an amino functionality siloxane if a short chain is used. It is because comparatively small addition of an amino functionality siloxane if a short chain is used. It is because character of optimal chain length and a hydroxy end ORGANO siloxane is determined by parameter of further others. One key parameter is compatibility with other ingredients of JIORUGANO siloxane polymer of an amino functionality siloxane, and an emulsion. Compatibility improves with chain length in whom a hydroxy end ORGANO siloxane increases, and the chemical similarity of various R substituents. For example, if a JIORUGANO siloxane is PDMS polymer substantially, it will be preferred to use short chain PDMS for improved compatibility. One with important compatibility is because it controls a partition of aqueous phase, a disperse phase containing JIORUGANO siloxane polymer, and an amino functionality siloxane of a between.

[0056]In a desirable mode, said amino functionality siloxane is formed by making Silang (IV) shown by amino functionality Silang (II), hydroxy end ORGANO siloxane (III), and $R^5{}_2SiG_{4-c}$ react. It is commercially available or this Silang is prepared by a publicly known method.

[0057]Substituent R^5 is chosen from a univalent hydrocarbon group which 1-15 carbon atoms were replaced individually, or is not replaced. An example of these bases is the same as what was given about the substituent R. Each R^4 is methyl preferably.

[0058]The substituent G is a basis of hydrolysis nature. A basis of hydrolysis nature useful for this invention is the same as having described X. The substituent G is an alkoxy group preferably and is methoxy or an ethoxy basis more preferably.

[0059]Inferior-letter c is 0, 1, or 2. c is 0 or 1 and c is 1 more preferably.

[0060]An example of Silang (IV) is the same as an example given upwards not only about dimethyldimethoxysilane, dimethyl diethoxysilane, ethyl methyl dimethoxysilane, and diisobutyl dimethoxysilane but about said cross linking agent. Preferably, it is alkoxysilane, this Silang has methyl trimetoxysilane and more preferred methyl triethoxysilane, and its methyl trimetoxysilane is the most preferred.

[0061]As for a hydrolytic basis on Silang, it is preferred that it is the same chemical nature as a hydrolytic basis on a cross linking agent. Therefore, as for a hydrolytic basis on Silang, when a hydrolytic basis on a cross linking agent is a methoxy group, it is preferred that it is an alkoxy group. But they do not necessarily need to be methoxy groups. Combination of two or more leaving groups which may give danger of immiscible nature or exothermic acid / base reaction should be avoided.

[0062]If ingredient (IV) was added to ingredient (II) and ingredient (III), storage life will have been improved about an amino functionality siloxane. If ingredient (II) and ingredient (III) are mixed, while viscosity of an amino functionality siloxane is the increase 7 - 28 days, gelling will take place, without making into less than one a mole ratio which is ingredient (II) opposite ingredient (III) and adding ingredient (IV). It depends for this actual time interval on quantity of added amino functionality Silang.

[0063]Ingredient (II), (III), and (IV) are mixed by various mole ratios, and an amino functionality

siloxane is formed. Usually, 1:1, even if there are few mole ratios of ingredient (II) and (IV) opposite ingredient (III), these ingredients are mixed so that it may be preferably set to at least 1.1:1, and a mole ratio of ingredient (IV) opposite ingredient (II) may be set to 0-2. The most desirable mole ratios of ingredient (II) opposite ingredient (III) are 0.2-0.8, and the most desirable mole ratios of ingredient (IV) opposite ingredient (III) are 0.2-0.8.

[0064]An amino functionality siloxane by mixing an ingredient containing amino functionality Silang (II) and hydroxy end ORGANO siloxane (III). Or it is formed mixing an ingredient which contains amino functionality Silang (II), a hydroxy end ORGANO siloxane, and Silang (IV) in a desirable example, and by making them react in each case. These ingredients are single kinds or are added as a mixture of a kind beyond 2 or it. This mixing is performed by blending these ingredients mutually at a room temperature, or other conventional methods which mix a compound. Although a mixed order is not important, it is preferred to mix ingredient (III) to ingredient (II) and to mix subsequently to ingredient (IV). Although ingredient (II), (III), and (IV) are net, are made to exist in [as a solution] material and it can mix, it is preferred to add them with net.

[0065]Said amino functionality siloxane is added to other ingredients of a silicone emulsion until a reaction between many ingredients is completed substantially. Quantity of time required for this is determined by experiment depending on a specific compound used. Typical reaction time is 5 to 24 hours. Supposing it wishes, this mixture can be heated in 50 ** - 70 **, and this can reduce reaction time in less than 2 hours.

[0066]An effective dose of an amino functionality siloxane is a quantity applied in order to make an improved adhesive property to a substrate by a silicone elastomer formed from a silicone emulsion, when water is removed. It depends for quantity of a required amino functionality siloxane on a molecular weight of a hydroxy end ORGANO siloxane. Generally an amino functionality siloxane on the basis of JIORUGANO siloxane polymer 100 weight section 0.10 - 10 weight-section **********, a required amino functional group of minimum density 0.0005 weight section -- an amino functional group (in each case, based on JIORUGANO siloxane polymer of 100 weight sections) of 0.005 weight sections is attained preferably -- I will come out. "An amino functionality siloxane of a weight section" used here means thing ** which multiplied the 1st amino group in an amino functionality siloxane, and wt% of the 2nd amino group by a weight section of an amino functionality siloxane in a compound on the basis of JIORUGANO siloxane polymer 100 weight section.

[0067]As for said amino functionality siloxane, a front stirrup of emulsification is added behind. Since when being added after an amino functionality siloxane's emulsifying ensures that the adhesive property of a silicone elastomer obtained when water is removed does not decrease, it is 2 to 5 hours preferably [adding an amino functionality siloxane within 8 hours after emulsification], and more preferably. It is preferred to add, before emulsifying an amino functionality siloxane for an ease of combination.

[0068]An ingredient (G) is acid and this is used as an optional component. Acid is believed to help for an amino functionality siloxane to entar into an emulsion. As another ingredient, this acid can be added as a part of other ingredients. For example, acetic acid will be formed when this DBTDA has already contained acetic acid when used with a solution when dibutyltin diacetate (DBTDA) is used as a condensation catalyst, or adding to a drainage system emulsion. In this invention, HCl, H_2SO_4, or organic acid, for example, carboxylic acid, can use any acid, for example. Carboxylic acid is preferred. That is because carboxylic acid and those corresponding amine salt act also as condensation cocatalyst with a tin condensation catalyst. As an example of carboxylic acid, there are acetic acid, formic acid, propionic acid, and crotonic acid. Acetic acid is the most desirable acid. Since carboxylic acid and those corresponding amine salt act as cocatalyst, when carboxylic acid is added, quantity of a tin condensation catalyst added can be reduced.

[0069]Usually, acid can apply quantity of 0.05 to 0.3 weight section on the basis of JIORUGANO siloxane polymer 100 weight section. Preferably, 0.06 to 0.2 weight section is added on the basis of JIORUGANO siloxane polymer 100 weight section, and this acid has 0.07 to 0.13 much more preferred weight section.

[0070]Said acid is added as a single kind or a mixture of a kind beyond 2 or it. This acid can add a front stirrup of emulsification behind. Adding is preferred before it will add them, if this acid has before bridge construction of JIORUGANO siloxane polymer therefore a tin condensation catalyst, and a required cross linking agent.

[0071]Since the performance characteristio of a silicone elastomer formed from a silicone emulsion or it of a certain kind is affected, additional arbitrary ingredients, for example, a filler, and other ingradients may be added with other ingredients according to a wish. As an example of a reinforoing filler and a filler for increase in quantity, there are calcium carbonate, a titanium dioxide, a zinc oxide, iron oxide, and kaolin olay. As an example of a filler which can be used in order to give fire retardancy or electric arc resistance, there are aluminum 3 hydrate, a fumed titania, and way acid zinc. Other arbitrary ingredients which include paints, stabilizer, or reinforcement nature resin in the spot can be added to this invention silicone emulsion.

[0072]An ingredient of an addition of all these should be examined in order that they may secure not having bad influence on the adhesive property of a silicone elastomer and storage life which are formed when a silicone emulsion or this emulsion of this invention is dried. The characteristic of said drainage system silicone emulsion and said silicone elastomer can give the characteristic of a wish by making it change and changing arbitrary ingredients of these additions.

[0073]Since improved adhesion to a substrate takes place regardless of a particle size, a particle size of a silicone emulsion does not restrict this invention. A particle size seems however, to exist in time stability of length with a useful silicone emulsion. This time is usually several months thru/or several years. Generally, a particle size of 200-1000 nm gives the stability of an emulsion of this level.

[0074]A silicone emulsion of this invention is formed by various methods. For example, this silicone emulsion can be prepared by a method of an emulsion polymerization currently taught to US-A 2891920, 3294725, 3355406, 3360491, and 3697469.

[0075]About an emulsion polymerization, an annular or linear siloxane oligomer is underwater distributed with a surface-active agent, and a reserve mixture is formed. Although an ampholytic surface active agent, an anionic surfactant, or a cationic surfactant is generally used, a mixture of an ampholytic surface active agent, an anionic surfactant or a cationic surfactant, and a nonionic surface active agent will also function. Subsequently, this reserve mixture is mixed by high shearing until an emulsion containing a disperse phase containing a glob of a with aqueous phase and a particle size of 100-5000 nm siloxane oligomer is formed. This mixing may take place also in what kind of type of commercial mixing device, and said mixed device is well-known at a person skilled in the art. In order to adjust pH, acid or a base may be added to said emulsion. or it may be added to a reserve mixture. Instead, said surface-active agent may be converted into a form of the acid or a base using an ionic exchange process as taught by US-A 3697469. Although a polymerization will proceed to satisfaction at a room temperature, it may carry out at a high temperature and desirable temperature requirements are 25 ** - 85 **. Probably, generally time of a polymerization is 1 to 24 hours depending on temperature and a molecular weight of a wish of polymer. After JIORUGANO siloxane polymer reaches a molecular weight of a wish, a polymerization is suspended by nautralizing this emulsion.

[0076]a cross linking agent (or [being required] — or — if wished) and a tin condensation catalyst can also be added before emulsification or after a polymerization. However, a cross linking agent and a tin condensation catalyst will often be added to an emulsion, after a polymerization finishes. This cross linking agent shifts into a disperse phase from aqueous phase in this case, and, in addition, must maintain that reactivity.

[0077]After said amino functionality silxane is added at any [between emulsion polymerizations] time, for example, a reserve mixture as some reserve mixtures before emulsification is emulsified, a front stirrup of a polymerization is added behind. As for it, when being added before said amino functionality siloxane's emulsifying, adding within 8 hours is preferred.

[0078]If a comparatively low-concentration polymer solid content is desired, water of an addition amount may be added in which stage of combination. A general polymer solid content is 20 to 75%. A desirable polymer solid content is 40 to 75%.

[0079]This acid may be independently added as a part of other ingredients, after a polymerization is completed. As a part of other ingredients, DBDTA will form acetic acid, for example, when it adds to water.

[0080]Arbitrary ones of other ingradients, for example, a filler, paints, stabilizer, reinforcement nature resin in the spot, etc. can be added to any time, after a polymerization is completed.

[0081]A desirable method of preparing a drainage system silicone emulsion is directly based on an emulsification method, and this is well-known and is taught to a person skilled in the art at US-A

4177177 or EP-A 0739947, 0739928, and 0739929.

[0082]A mixture of JIORUGANO siloxane polymer by which praforming was carried out, a surface-active agent, and water is emulsified sufficient period and by mixing by shearing sufficient at temperature of 10 ** - 70 ** about direct emulsification. In this method, useful JIORUGANO siloxane polymer is characterized as what has the viscosity 5000 - 500,000 mPa-s at 25 **. However, supposing viscosity is prepared using a solvent, a polymer blend, etc., polymer of a comparatively high molecular weight can be used.

[0083]Generally, an ampholytic surface active agent, an anionic surfactant, a cationic surfactant, or a nonionic surface active agent is used as independent or a mixture. This mixing takes place in a commercial mixing device of one of types. This mixed device is well-known at a person skilled in the art.

[0084]A cross linking agent (when required, or when it is wished) and the tin condensation catalyst can add a front stirrup of emulsification after emulsification. However, said cross linking agent and a tin condensation catalyst will often be added before emulsification. If it adds after emulsification, this cross linking agent must shift into a disperse phase from water, and must still maintain that reactivity.

[0085]Said amino functionality siloxane can be added even in front of emulsification or in the back at any time. When being added after this amino functionality siloxane's emulsifying, it is preferred to add within 8 hours after bridge construction of an emulsion.

[0086]When a low polymer solid content is desired, water of an addition amount can also be added in one stage of the combination. A general polymer solid content is 10 to 98%. A desirable polymer solid content is 20 to 85%, and is much more desirable. [40 to 85% of]

[0087]Acid can also be added at any time as a part of other another or ingredients (namely, DBDTA will form acetic acid at the time of addition of water, for example). In a desirable method, carboxylic acid and an amino functionality siloxane are added before bridge construction. If this wishes, it will make it possible to reduce quantity of a tin condensation catalyst.

[0088]Any arbitrary ingredients can add a filler, paints, stabilizer, resin for reinforcement in the spot, etc. in any stages of combination, for example.

[0089]In a more desirable method, it is JIORUGANO siloxane polymer (here) of 100 weight sections.

X is hydroxyl, n is 2 and Y is Si. R and R^1 is a methyl group, respectively — an amino functionality siloxane of dibutyltin diacetate catalyst:1 weight section of polyoxyalkylene alkyl ether surface-active agent:0.06:3 copy weight section [water:2 copy] (this) Amino functionality Silang (II)$(CH_3O)_3Si$-$(CH_2)_3$-NH-$(CH_2)_2$-NH_2 Hydroxy end ORGANO siloxane (III)HO-$(Si(CH_3)_2)_n$-OH (here) It is formed by making Silang (IV) methyl trimetoxysilane react, and b is 7-9. And an amino functional group of 0.005 weight sections exists, and a mole ratio of ingredient (II) and ingredient (IV) opposite ingredient (III) is at least 1 again, A mole ratio of ingredient (IV) pair ingredient (II) is two or less. Mix and acetic acid of 0.1 copy under high shearing, and form a high solid concentration emulsion, and it dilutes so that this high solid concentration emulsion may be desired with water, Subsequently, by constructing a bridge in this emulsion, a drainage system silicone emulsion is formed by adding an isobutyl TORIMETOSHI xylan cross linking agent of 0.8 weight sections.

[0090]After removing water, a silicone elastomer formed from this invention silicone emulsion has an improved adhesive property to a substrate. A silicone emulsion of this invention has long storage life compared with the conventional emulsion.

[0091]

[Example]The following examples are shown in order to explain the constituent of this invention further. In the following examples, unless it refused in particular, one day after making an emulsion, the cast of the humid emulsion was carried out to the film, and it examined, after drying these films for seven days.

[0092]The result of the durometer was obtained by the method indicated to ASTM C661 "IndentationHardness of Elastomeric-Type Sealantby Means of a Durometer." The result of tension and elongation, The size of L the dumbbell specimen which is 1.27 mm. It obtained by the method indicated to "Vulcanized Rubberand Thermoplastic Rubbers and Thermoplastic Elastomers-Tension" which used and was indicated to ASTM D412. The Shore A hardness value was obtained by the method indicated to ASTM C661 "IndentationHardness of Elastomeric Type Sealants by Means

of a Durometer."

[0093]"Me" used here means methyl, "Et" means ethyl, and "laboratory conditions" is temperature **2 **, and 50% of relative humidity **5% of meanings of 23 **.

[0094](A) The adhesive valuation method sample was formed on various substrates to the bead (bead) 50 mm in length, and 18 mm in width. The adhesive property was evaluated by putting slitting into a bead using a laser braid by sealant / substrate interface, and pulling this bead by hand at the angle of 45 degrees to a level base material surface. When a bead caused cohesive failure, the adhesive property was graded "easily." The bead caused adhesive failure, and when power equivalent to the time of removing a bead from a base material surface was required, the adhesive property was graded with "good." When adhesive failure was caused by the exfoliation power in which a bead is comparatively low, it graded with "**."

[0095](Example 1) Hydroxy end polydimethyl siloxane (PDMS) polymer of 5000 copies (viscosity is 50 Pa-s at 25 **). The 10-l. Turello™ mixer was loaded with the Tergitol(trademark) TMN-deionization (D) water of 6 (it is ethoxylation trimethyl nonanol with a nonionic surface active agent), and 100 copies of 100 copies.

[0096]This mixer was stirred under the vacuum for 5 minutes using the distributed braid revolving at 1200 rpm. After mixing, when it observes, it is transparent and is the gel (CHIKISOTO rope) of non fluidity, and formation of the o/w emulsion of high solid concentration was shown. The distributed braid was rotated at 600 rpm, stirring was begun again, and DI water of 250 copies was added. applying a vacuum and rotating a distributed braid at 1200 rpm — an emulsion — further — it stirred for 2 minutes. When observed, the white emulsion looked like milk thin time. The distributed braid was rotated at 600 rpm, stirring was begun again, and DI water of 1000 copies was divided into two equal portions, and was added. The vacuum was applied and this mixture was stirred during [2 minute] these addition. The o/w emulsion mixed that it is white and well like milk was formed, and this had neither a lump nor gel. To this emulsion. In the hydroxy end PDMS fluid (the degrees of polymerization 7-9, viscosity of 25 ** 0.04 Pa-s) of 74.6 copies, the N-(2-aminoethyl)-3-aminopropyl trimethoxysilane (AEAPTMS) of 15.2 copies, and the methyl TORIMETOSHI xylan (MTM) of 10.2 copies. The obtained amino functionality siloxane (AFF2) of 50 copies was added by carrying out cold blending. The distributed braid was rotated at 600 rpm, stirring of this mixture was started, the vacuum was applied, and this mixture was stirred for 2 minutes. subsequently — adding IBTMS (isobutyl trimethoxysilane) of 40 copies — the bottom of a vacuum — a mixture — further — it stirred for 2 minutes. Next, DBTDA (dibutyltin diacetate) of ten copies was added and this mixture was stirred for 2 minutes using the distributed braid rotated at 600 rpm under a vacuum. The Semco (trademark) cartridge was filled up with this silicone emulsion that constructed the bridge, and all the air which centrifuged this and was caught was removed.

[0097]After aging this silicone emulsion which constructed [above-mentioned] the bridge at a room temperature for 18 hours, the sample was formed on the 2.54 mm (100 mils)-thick film, and it was made to harden under a laboratory condition for seven days. The tensile strength of this hardened translucent silicone elastomer was 0.5MPa (80 psi), 2000% of the maximum elongation, and the Shore A durometer 10. On the substrate with which 16 differed, other samples of this aged silicone emulsion were formed to a bead 50 mm in length, and 18 mm in width, and will be stiffened under laboratory conditions for seven days. [available to one commercial target because of comparison] Calcium carbonate. The latex sealant (Dow Corning(trademark) Silicone Plus) and the RTV alkoxy hardenability silicone sealant (General Electric(trademark) GEII) of one commercially available hygroscopic-surface-moisture hardenability (non-latex) with which it was filled up. It evaluated under the same condition. Table 1 shows an adhesive result.

[0098]

[Table 1]

Example of sealant adhesiveness substrate 1Silicone PlusGE II ceramic tile A . ** A DEYURANA (Duranar) ** . Good Right concrete A Good Right Korean (Corian). ** Good An A paint spreading pine (alkyd paint). A A An A paint spreading pine (latex paint). A A A United States Japan cedar A A A alumite aluminum . A A A polyvinyl chloride (unplasticized) A Good A glass A Good A brass A A **** A A A polycarbonate ** Good ** stainless steel A A Right mortar A Good Right PMMA (polymethylmethacrylate) ** Good **[0099](Example 2)

(a) The viscosity in 25 ** loaded the Whip Mix™ pot of 350mL with 100 copies of hydroxy end PDMS

polymer of 50 Pa-s, and one copy of amino functionality siloxane (AFF2) prepared in Example 1, and stirred it for 30 seconds under the vacuum. Subsequently, DBTDA of 0.06 copy was added and this mixture was stirred for 30 more seconds under the vacuum. Glacial acetic acid of 0.1 copy was added and this mixture was stirred for 30 more seconds under the vacuum. Next, Tergitol(trademark) TMN-6 (the nonionic surface active agent, the ethoxylation trimethyl nonanol) and DI water of three copies of two copies were added, this mixture was stirred for 30 more seconds under the vacuum, and the o/w emulsion of high solid content of a little transparent non fluidity was obtained. Subsequently, stirring for 30 seconds was performed under the vacuum between addition [water / DI / portions / four / of four copies each], adding IBTMS of 0.8 copy — this mixture — further — it stirred for 30 seconds. The Semco (trademark) cartridge was filled up with this silicone emulsion, and all the air which centrifuged this and was caught was removed. After aging at a room temperature for 18 hours, the sample was formed on the 2.54 mm (100 mils)-thick film, and it was made to harden under a laboratory condition for seven days. The tensile strength of this hardened translucent silicone latex elastomer was 0.25MPa (40 psi), 1570% of the maximum elongation, and the Shore A durometer 6. On the glass as a substrate, concrete, the pine that carried out the painting cloth, and a U.S. Japan cedar, other samples of the silicone emulsion aged for 18 hours were formed to a bead 50 mm in length, and 18 mm in width, and will be stiffened under laboratory conditions for 14 days. As the adhesive property of said silicone latex was indicated above (A), it was evaluated. said latex was excellent in all the substrates — it ********(ed) (cohesive failure). After carrying out storage aging of said damp silicone emulsion for four weeks under a laboratory condition, as the sample of other silicone emulsions was shown above (A), it was examined about the adhesive property. Said latex showed the adhesive property excellent in all the substrates (cohesive failure).

[0100](b) An amino functionality siloxane was not added at all, and also the same procedure as Example 2 (a) was followed. The tensile strength of this hardened translucent silicone elastomer was 0.6MPa (86 psi), 1080% of the maximum elongation, and the Shore A durometer 11. This elastomer showed the **** adhesive property to glass, and showed the good adhesive property to concrete, the paint spreading pine, and the U.S. Japan cedar. After carrying out storage aging of said damp silicone emulsion for four weeks under a laboratory condition, as the sample of further others of the above-mentioned emulsion was indicated above (A), it was examined about the adhesive property. This sealant showed the **** adhesive property to glass, and showed the good adhesive property to concrete, the paint spreading pine, and the U.S. Japan cedar.

[0101](c) 3-aminopropyl triethoxysilane (APTES) of 0.152 copy was transposed to said amino functionality siloxane, and also this example was prepared according to the procedure of Example 2 (a). The tensile strength of this hardened translucent silicone elastomer was 0.32MPa (46 psi), 1280% of the maximum elongation, and the Shore A durometer 6. This elastomer showed the adhesive property outstanding to glass, concrete, the paint spreading pine, and the U.S. Japan cedar. After carrying out storage aging of said damp silicone emulsion for four weeks under a laboratory condition, as the sample of further others of said silicone emulsion was indicated above (A), it was examined about the adhesive property. This sealant showed the **** adhesive property to glass, and showed the good adhesive property to concrete, the paint spreading pine, and the U.S. Japan cedar.

[0102](Example 3) The 100mL mixer cup was loaded with hydroxy end PDMS polymer of 50 copies of viscosity 50 Pa-s in 25 **, and DBTDA of 0.03 copy, and it stirred for 10 seconds in the Hauschild™ mixer. Subsequently, 0.5 copy of amino functionality siloxane (AFF2) of Example 1 was added, and this mixture was stirred for 10 more seconds in this mixer. Subsequently, IBTMS of 0.375 copy was added and this mixture was stirred for 10 more seconds. subsequently — adding Tergitol(trademark) TMN-6 surface-active agent of one copy, and 1.5 copies of 0.072 mol of DI underwater glacial-acetic-acid solution — this mixture — further — it stirred for 10 seconds and the high solid content o/w emulsion was formed. 0.072-mol acetic acid of three copies, three copies, and four copies was added continuously, and this emulsion was further diluted by stirring this mixture for 10 seconds by the mixer between addition. This silicone emulsion was a white substance like milk, moved this to the Semco (trademark) cartridge, and removed all the air which centrifuged this and was caught. After aging at a room temperature for 18 hours, the sample was formed on the 2.54 mm (100 mils)-thick film, and it was made to harden under a laboratory condition for seven days. The tensile strength of this hardened translucent silicone elastomer was 0.34MPa (50 psi), 1290% of the maximum elongation, and the Shore A durometer 6. On the glass as a substrate, concrete, the pine that carried out the

painting cloth, and a U.S. Japan cedar, other samples of the silicone emulsion aged for 18 hours were formed to a bead 50 mm in length, and 18 mm in width, and will be stiffened under laboratory conditions for 14 days. Said sealant showed the adhesive property excellent in all the examined substrates (cohesive failure).

[0103](Example 4) Two or more amino functionality siloxanes of different siloxane chain length were prepared by carrying out cold blending of AEPTMS, the hydroxy end ORGANO siloxane (siloxane diol), and MTM which were distilled in a polyethylene container. This mixture was made to react for 24 hours, and when measured with that blasting-fumes liquid chromatography (GLC), 100% of the hydroxy functional groups of the hydroxy end ORGANO siloxane had disappeared. Table 2 shows the ratio by amino functionality Silang, various chain length's siloxane diol, and the weight section that blended MTM. Each siloxane diol consists of siloxane diol of the chain length from whom a certain distribution differed, and those number average molecular weights correspond to DP (degree of polymerization) 4, 8, and 39. Subsequently, it assumed and calculated that the above-mentioned DP and siloxane diol:AEAPTMS:MTM were 0.122:0.068:0.075 about the mixture ratio reported to Table 2.

[0104]

(表2)

	アミノ官能性シロキサン		
成分 (部)	AFF1	AFF2	AFF3
シロキサンジオール DP=4	60.2		
シロキサンジオール DP=8		74.6	
シロキサンジオール DP=39			93.3
素製した AEAPTMS	23.8	15.2	4
MTM	16	10.2	2.7

[0105]Said amino functionality siloxane (AFF2) is an amino functionality siloxane used in all other examples that include AFF2 in Examples 1 and 2.

[0106]Respectively, three elastomeric compositions were prepared using one of the amino functionality siloxanes of these. The addition of AFF1 and AFF3 was based on the amount of amino functionality Silang of equimolar on the basis of one weight section of AFF2 as reference. In each combination, the Whip Mix™ mixer of 350mL was loaded with hydroxy end PDMS polymer (viscosity 50 Pa-s in 25 **) of 100 copies and AFF2 of 1 or 1 copy of AFF of 0.64 copy, or AFF3 of 3.8 copies, and each mixture was stirred for 30 seconds under the vacuum. DBTDA of 0.06 copy and IBTMS of 0.75 copy were added to these mixtures, it is under a vacuum and also this mixture was stirred for 30 seconds. adding glacial acetic acid of 0.10 copy — this mixture — further — it stirred under a 30-second vacuum. subsequently — adding Tergitol(trademark) TMN-6 surface-active agent of two copies, and DI water of three copies — this mixture — further — it stirred for 30 seconds and the o/w emulsion of high solid content was formed, respectively. By adding continuously five portions of DI water of four copies each, these emulsions were diluted further and stirring for 30 seconds was performed between addition and under the vacuum. All the air that moved these silicone emulsions to the Semco (trademark) cartridge, centrifuged, and was caught was removed. After aging at a room temperature for 18 hours, the sample of each combination was formed on the 2.54 mm (100 mils)-thick film, and it was made to harden under a laboratory condition for seven days. If attached to the physical-properties data of these sealant compound, refer to the 2nd (b) table.

[0107]

[Table 3]

the — 2 front [(b)] sealant compound physical-properties S-AFF1 S-AFF2 S-AFF3 tensile strength (MPa) — 0.41 0.28 The 0.35 maximum elongation (%) 2570 1480 730 Shore (A) durometer 2 3 3[0108] On glass, other samples of the silicone emulsion aged for 18 hours were formad to a bead 50 mm in length, and 18 mm in width, and will be stiffened under laboratory conditions for 14 days. All the samples showed the adhesive property outstanding to glass (cohesive failure).

[0109](Example 5) Two or more amino functionality siloxanes were prepared by carrying out cold blending of siloxane diol (viscosity 0.04 Pa-s in 25 **) and MTM of amino functionality Silang and DP 7-9 which have the 1st, 2nd, and 3rd amino groups in a polyethylene container. This mixture was made to react for 24 hours, and when measured by GLC after that, 100% of -OH functional groups of siloxane diol had disappeared. :(a) APTES and $H_2N-(CH_2)_3Si(OC_2H_5)_3$ which evaluated the following amino functionality Silang (primary amine), (b) AEAPTMS (1st and 2nd amine group) and (c) bis (trimethoxysilylpropyl)amine $(CH_3O)_3Si(CH_2)_3NH(CH_2)_3Si(OCH_3)_3$ (the 2nd amine) and (d) N - [3-(triethoxy silyl) propyl] -4,5-dihydroimidazole, $(C_9H_{20}O)_3SiCH_2)_3$ - [N-CH=N-CH_2-CH_2] - (The -N-CH=N-CH_2-CH_2-portion of a molecule is an annular group here) . (tertiary amine). The 3rd table shows the ratio by a weight section when various amino functionality Silang, hydroxy end siloxanes, and MTM are blended.

[0110]

[Table 4]

3rd table amino functionality siloxane component (part) AFF2AFF4AFF5AFF6 siloxane diol DP=8 74.6 74.6 69.06 72.1 (a) 15.2 (b) 15.2 (c) 21.5 (d) 18.04MTM. 10.2 9.44 9.86 * — it is as having defined (a), (b), (c), and (d) here above.

[0111]Each compound contained one of amino functionality siloxans AFF2, AFF4, AFF5, or AFF6 copies, and also four compounds wera prapared acoording to tha procedure of the above-mentioned example 4. Subsequently, as it was filled up with this silicone emulsion and had indicated for Example 4, it examined about tha adhesive proparty. The sample which blended AFF2 or AFF4 showed cohesive failure mode to glass, and the sample which blended AFF5 or AFF6 showed adhesive failure mode to glass.

[0112](Example 6) The amino functionality siloxane was prepared by carrying out cold blending of the AEAPTMS by one the half of the quantity used into AFF2 of Example 4, twice, or 3 times the molar quantity of this. In the polyethylene container, MTM was added to the hydroxy end PDMS ORGANO siloxane (viscosity 0.04Paands in 25 **) (siloxane diol) of DP 7-9. This mixture was made to react for 24 hours, and when measured by GLC after that, 100% of -OH functional groups of siloxane diol had disappeared. The 4th table shows the presentation of the plurality of the prepared amino functionality siloxane.

[0113]

[Table 5]

4th table amino functionality siloxane AFF2 AFF7. Nine ingredients (part) (1x mol) (1 / 2x mol) (2x mol) (3x mol) of AFF8 AFF siloxane diol DP=8 74.6 80.74 64.76 57.21AEAPTMS 15.2 8.23 26.39. 34.97MTM 10.2 11.03 8.85 7.82[0114]Four compounds were prepared using one of above-mentioned amino functionality siloxane AFF2, AFF7, AFF8, and AFF9. In addition, the addition to the silicone emulsion of AFF2, AFF7, AFF8, and AFF9 was kept constant as one copy on the basis of 100 copies of PDMS polymer. As it was prepared and filled up with these compounds and had indicated for Example 4, it examined about the adhesive property. The sample which blended AFF2, AFF8, or AFF9 showed cohesive failure mode to glass, and the sample which blended AFF7 showed adhesive failure mode to glass.

[0115]Equivalent weight of AFF2 and AFF7 were mixed, the 0.75x mol (AFF2/7) was formed, and the experiment of further others was conducted by adding one copy of this blend on the basis of 100 copies of PDMS polymer in a compound. This elastomer was hardened, and as it indicated above, when it examined above, cohesive failure was shown to glass. The minimal dose of the amino functional group required in order that this experiment may obtain the cohesive failure to glass showed that it was a $>5x10^{0}5x10^{-4}$ part on the basis of 100 copies of PDMS polymer.

[0116]After neglecting it for 18 hours, the sample was formed on the 2.54 mm (100 mils)-thick film, and physical properties were acquired about the compound which will stiffen this subsequently to the bottom of a laboratory condition for 14 days. The 5th table shows the character of various compounds.

[0117]

[Table 6]

5th table Si -RANTO ** ** Adhesive property to thing physical-properties S-AFF2 S-AFF7 S-AFF8

S-AFF9 S-AFF2/7 glass Condensation Condensation Condensation Condensation Condensation durometer 5 8 2 0 it pulls six (MPa) -- 0.32. 0.36 0.17 0.12 The 0.34 maximum elongation (%) 1280 1480 1840 2480 1280[0118]The amino functionality Silang contant in an amino functionality siloxane takes for increasing, a durometer and tensile strength decrease, and the maximum elongation of an elastomer increases so that clearly from the 5th table. If there is much quantity of amino functionality Silang (added via an amino functionality siloxane) in a compound, when it is a dry INGU down (drying down), the comparatively long induction period before an elastomer hardens will be brought about (till the 10th). Therefore, a lot of amino functionality Silang is not desirable.

[0119]In order to measure the stability of various amino functional fluids of Example 6, the sample of each 15-g amino functionality siloxane was put in the polyethylene bottle, and it purged with nitrogen, and stored into a 50 ** furnace. AFF7 with the lowest quantity of amino functionality Silang was gelled by the storage on less than the 7th. Covering the storage time on the 30th about the remaining amino functionality siloxane, gelling did not take place.

[0120](Example 7) In the polyethylene container, two or more amino functionality siloxanes were prepared by carrying out cold blending of siloxane diol (viscosity 0.04 Pa-s in 25 **) and MTM of DP 7-9 to AEAPTMS, APMDES, or APTES. This mixture was made to react for 24 hours, and when measured by GLC after that, 100% of -OH functional groups of siloxane diol had disappeared. The 6th table shows the presentation of the prepared amino functionality siloxane.

[0121]

[Table 7]

6th table amino functionality siloxane component (part) AFF2AFF10AFF11 siloxane diol DP=8 74.6 76.2 74.67AEAPTMS 15.2 -- -APMDES - 13.38 -APTES. - - 15.12MTM 10.2 10.42 10.21[0122]AFF2, AFF10, and AFF11 were evaluated in the elastomeric composition, respectively. In each case, the addition of AFF2, AFF10, or AFF11 was kept constant as one copy on the basis of 100 copies of PDMS polymer. As it was prepared and filled up with these compounds and had indicated for Example 4, it examined about the adhesive property. All the blended samples showed cohesive failure mode to glass.

[0123]In order to measure the stability of AFF2 of Example 7, AFF10, and AFF11, the sample of each 15-g amino functionality siloxane was put in the polyethylene bottle, and it purged with nitrogen, and stored into a 50 ** furnace. AFF10 which is the amino functionality siloxane prepared using APMDES was gelled by the storage for less than four weeks. About AFF2 and AFF11, the same storage time was covered and gelling did not take place.

[0124](Example 8) AEAPTMS of the molar quantity used about AFF2 in Example 4 in the polyethylene container. By carrying out cold blending of siloxane diol (viscosity 0.04 Pa-s in 25 **) and alkoxysilane of DP 7-9, two or more amino functionality siloxanes were prepared. : using the following alkoxysilane -- MTM, tetraethyl orthosilicate (TEOS), and n-propyl orthosilicate (NPOS). These mixtures were made to react for 24 hours, and when measured by GLC after that, 100% of -OH functional groups of siloxane diol had disappeared. The 7th table shows the presentation of the prepared amino functionality siloxane.

[0125]

[Table 8]

7th table amino functionality siloxane component (part) AFF2AFF10AFF11 siloxane diol DP=8 74.6 71.59 68.07AEAPTMS 15.2 14.59 13.87MTM 10.2 - -TEOS. - 13.82 -NPOS - - 18.02[0126]AFF2, AFF12, and AFF13 were evaluated in the elastomeric composition, respectively. In each case, the addition of AFF2, AFF12, or AFF13 was kept constant as one copy on the basis of 100 copies of PDMS polymer. As it was prepared and filled up with these compounds and had indicated for Example 4, it examined about the adhesive property. All the blended samples showed cohesive failure mode to glass. These elastomers were examined also about freeze thaw stability, and it found out passing in 10 times of ASTM **** cycles.

[0127](Example 9) AEAPTMS of the molar quantity used about AFF2 in Example 4 in the polyethylene container. By carrying out cold blending of siloxane diol (viscosity 0.04 Pa-s in 25 **) and the alkoxy OKISHIMO silane of DP 7-9, two or more amino functionality siloxanes were prepared. : using the following alkoxysilane -- MTM, tetraethyl orthosilicate (TEOS), and n-propyl orthosilicate (NPOS). This alkoxy OKISHIMO silane mixture 3% of tetra (methylethyl keto KISHIMO) Silang (TOS), 84% of tri- and dioxy MOSHIRAN, 10% of mono- OKISHIMO silane (other bases on a molecule are ethoxy

bases here), and 2% of methylethyl keto KISHIMO silane solvent were included. This mixture was made to react for 24 hours, and when measured by GC after that, 100% of -OH functional groups of siloxane diol had disappeared. AFF2 was prepared. The 8th table shows the presentation of the prepared amino functionality siloxane.

[0128]

(表9)

第8表

アミノ官能性シロキサン

アミノ官能性シロキサン化合物 (部)	AFF2	AFF14
シロキサンジオール DP=8	74.6	67.88
AEAPTMS	15.2	13.83
MTM	10.2	-
TOS	-	18.19

[0129]AFF2 and AFF14 were evaluated in the elastomeric composition, respectively. In each case, the addition of AFF2 or AFF14 was kept constant as one copy on the basis of 100 copies of PDMS polymer. As it was prepared and filled up with these compounds and had indicated for Example 4, it examined about the adhesive property. All the blended samples showed cohesive failure mode to glass. These were examined also about freeze thaw stability and it found out passing in 10 times of ASTM **** cycles.

[0130](Example 10) Prepared the following cold blends in the polyethylene container, it was made to react for 24 hours, and the effect of the 2 premix thing of two or more ingredients containing an amino functionality siloxane was evaluated.

(a) What blended AEAPTMS with siloxane diol (DP 7-9, 25 ** viscosity 0.04 Pa-s);

(b) What blended AEAPTMS with what [blended MTM with siloxane diol (DP 7-9, 25 ** viscosity 0.04 Pa-s)], and (c) MTM.

[0131]The 9th table shows the presentation of prepared various functional fluids.

[0132]

[Table 10]

The 9th table official Ability Sex Style 16 ingredients (part) of body AFF15 F1 AFF(a)(a) (b) (c) siloxane diol DP=8 83.07 87.97 -AEAPTMS 16.93 - 59.84MTM - 12.03 40.16[0133]GLC analysis of said functional fluid showed that MTM and siloxane diol of (b) had not reactad. Three compounds which use one of said the functional fluids, respectively were prepared and evaluated.

[0134]The mixture obtained about each compound by adding hydroxy end PDMS polymer (viscosity 50 Pa-s in 25 **) of 200 copies, Tergitol(trademark) TMN-6 surface-active agent of four copies, and DI water of four copies to the Whip^{TM} mixture of 350mL was stirred for 30 seconds under the vacuum. DI water of 50 copies was divided into five portions whose number is ten, respectively, and was added to these high solid content emulsions. To these diluted emulsions, AFF15 (a) of 1.8 copies, adding F1 (b) of 1.7 copies, or either (the quantity of these gives AFF2 of two copies of mol equivalents) of AFF16 (c) of 0.51 copy with IBTMS of 1.6 copies, and DBTDA of 0.4 copy -- this mixture -- the bottom of a vacuum -- further -- it stirred for 30 seconds. As it was filled up with these silicone emulsions and indicated for Example 4, it examined about the adhesive property. The sample prepared using AFF15 (a) showed cohesive failure to glass, and the sample prepared using F1 (b) or AFF16 (c) on the other hand showed adhesive failure to glass. The character of these elastomers is shown in the 10th table.

[0135]

[Table 11]

adhesive property to compound nature S-AFF15 S-F1 S-AFF15 glass of the 10th table sealant

Condensation Condensation a condensation durometer (Shora A) -- 6 12 10 tensile strength (MPa) - - 0.47 0.65 The 0.55 maximum elongation (%) 1600 880 1280[0136]In order to measure the stability of various functional fluids, the sample of each 15-g fluid was put into the polyethylene bottle, and it

purged with nitrogen, and stored into a 50 ** furnace. AFF15 (amino functionality siloxane prepared without using MTM) was gelled within four weeks of storage. About F1 and AFF16, no gelling also took place to the same storage time.

[0137](Example 11) In the polyethylene container, cold blending of siloxane diol (viscosity 0.04 Pa-s in 25 **), MTM, and the functionality ORGANO siloxane of DP 7-9 was carried out, and the amino functionality siloxane was prepared by making this mixture react for 24 hours. :(a) AEAPTMS which uses the following functionality ORGANO siloxanes, (b) Gamma-glycidoxypropyltrimetoxysilane (GPTMS), (c) gamma-methacryloxpropyl trimethoxy silane (MAPTMS), and (d) gamma-mercapto propyltrimethoxysilane (MPTMS) were used. Various functionality ORGANO siloxanes were added to said cold blend by the fixed mole ratio. Table 11 shows the presentation of various functional fluids.

[0138]

[Table 12]

Two F2F3F4 ingredients (part) of the 11th table amino functionality siloxanes AFF (a) (b) (c) (d) Siloxane diol DP=8 74.6 74.6 74.6 74.6AEAPTMS 15.2 - - -GPTMS - 16.2 - -MATMS - - 17 - MPTMS - - - 13.4MTM 10.2 10.2 10.2 10.2[0139]Each of these functional fluid was evaluated in the elastomeric composition. The addition to the silicone latex compound of a functional fluid was kept constant as one copy on the basis of 100 copies of PDMS polymer. As it was prepared and filled up with these compounds and had indicated for Example 4, it examined about the adhesive property. The sample prepared using AFF2 (a) showed cohesive failure to glass, and the sample prepared using F2 (b), F3 (c), or F4 (d) showed adhesive failure to glass.

[0140](Example 12) In the polyethylene container, two or more amino functionality siloxanes were prepared by carrying out cold blending of AEAPTMS, MTM, and the hydroxy end ORGANO siloxane. DP 7-9 indicated for the example 1 of :(a) using the following siloxane diol, the PDMS ORGANO siloxane which has viscosity 0.04 Pa-s in 25 **, (b) an average of [average DP 5-7 and] -- an average of [the polyphenyl MECHIRUSHIROKISAN ORGANO siloxane of 6.55% of -OH content and (c) average DP 5-7, and] -- the PORITORIFURUORO propylmethyl ORGANO siloxane of 6.10% of -OH content. These mixtures were made to react for 24 hours, and when measured by GLC after that, 100% of -OH functional groups of the hydroxy end ORGANO siloxane had disappeared. The 12th table shows the presentation of prepared various amino functionality siloxanes.

[0141]

[Table 13]

12th table amino functionality siloxane component (part) AFF2AFF17AFF18 dimethylsiloxane diol 74.6 - - -phenylmethyl siloxane diol - 64.77 - trifluoropropylmethylsilanediol . - - - 66.38AEAPTMS 15.2 21.08 20.12MTM 10.2 14.5 13.5[0142]Each of these amino functionality siloxane was evaluated in the elastomeric composition. The addition to said compound of an amino functionality siloxane was kept constant as one copy on the basis of 100 copies of PDMS polymer. As it was prepared and filled up with these compounds and had indicated for Example 4, it examined about the adhesive property. The sealant sample prepared using AFF2 (a) showed cohesive failure to glass, wood, and concrete, and showed mixed destructive mode (50% of cohesive failure) to aluminum of mill finishing. The sample prepared using AFF17 or AFF18 showed adhesive failure to glass and mill finishing aluminum, and showed cohesive failure to wood and concrete. The physical properties of all the samples are dramatically alike about the durometer, the Shore range of 4-5 is shown, and tensile strength is 0.34 - 0.37MPa.

The maximum elongation was 2060% - 2300%.

[0143](Example 13)

(a) The following experiments were conducted in order to prove the stability of the reserve mixture which consists of an amino functionality siloxane (AFF2), IBTMS, hydroxy end JIORUGANO siloxane polymer, and DBTDA. The 10-l. Turello™ mixer was loaded with :5000 copy hydroxy end PDMS polymer (25 ** viscosity 50 Pa-s) and AFF2 of 50 copies which prepared said reserve mixture as follows. This mixture was stirred for 30 seconds under the vacuum. adding IBTMS of 40 copies, and DBTDA of three copies to this mixture -- this mixture -- the bottom of a vacuum -- further -- it stirred for 30 seconds. When the viscosity of this mixture was observed, there was no big increase in viscosity for 4 hours. 104 g of this reserve mixture was taken at intervals of 60 minutes, and Whip Mix™Mixer of 350mL was loaded. When Tergitol(trademark) TMN-6 surface-active agent of two

copies and DI water of two copies were added to each of these samples and this mixture was stirred for 30 seconds under the vacuum, it was slightly translucent and the o/w emulsion of high solid content was obtained with non fluidity. Subsequently, DI water was stirred for 30 seconds under the vacuum between each addition [portions / four / of four copies each]. The sample taken from 1, 2, 3, and the reserve mixture of 4 hours after was emulsified satisfactorily at all, and the obtained elastomer showed cohesive failure to glass.

(b) Other experiments were conducted, in order to evaluate whether the amino functionality siloxane has been added however for a long time after emulsifying. Hydroxy end PDMS polymer (25 ** viscosity 50 Pa-s) of 5000 copies, IBTMS of 40 copies, and DBTDA of three copies were added, and this mixture was stirred for 30 seconds under the vacuum. Tergitol(trademark) TMN[of 100 copies]-6 was added to this mixture, and this mixture was stirred for 30 seconds under the vacuum. Subsequently, when DI water of 150 copies was added and this mixture was stirred under the vacuum for 5 minutes, it was slightly translucent and the o/w emulsion of high solid content was obtained with non fluidity. 106 g of this silicone latex dispersing element was taken at intervals of [regular] 60 minutes. Whip Mix™Mixer of 350mL was loaded with these samples, and AFF2 of one copy was added. Subsequently, this mixture was stirred for 30 seconds under the vacuum. To this high solid content silicone emulsion, DI water was divided into four portions of four copies, respectively, and it added, and stirred for 30 seconds under the vacuum between each addition. The amino functionality siloxane could be added after emulsification of other ingredients of all the till 8 hours, and, in addition, giving the cohesive failure of the hardened sealant to glass was found out.

[0144]The Whip Mix™ pot of 350mL was loaded with hydroxy end PDMS polymer (viscosity of 25 ** 50Pa, ands) of 95.67 copies, and DBTDA of 0.06 copy, and this mixture was stirred for 30 seconds under the vacuum. adding AFF2 of 0.93 copy to this mixture -- this mixture -- the bottom of a vacuum -- further -- it stirred for 30 seconds. subsequently -- adding glacial acetic acid of 0.08 copy -- this mixture -- the bottom of a vacuum -- further -- it stirred for 30 seconds. IBTM of 0.70 copy was added to this mixture, and this mixture was stirred for 30 seconds under the vacuum. adding Tergitol(trademark) TMN-6 and DI water of 2.32 copies of 2.06 copies to this mixture -- this mixture -- the bottom of a vacuum -- further -- when stirred for 30 seconds, it was slightly translucent and the o/w emulsion of high solid content was obtained with non fluidity, adding 66.28 copies of underwater kaolin viscosity dispersing elements of 70% of solid content, and 44.9 copies of dispersing elements of the underwater rutile titanium dioxide of 62% of solid content to this mixture - - this mixture -- the bottom of a vacuum -- further -- it stirred for 30 seconds. All the air that filled up the Semco (trademark) cartridge with this silicone emulsion, centrifuged, and was caught was removed. After aging this emulsion at a room temperature for 18 hours, the sample of each combination was formed on the 2.54 mm (100 mils)-thick film, and it was made to harden under a laboratory condition for 14 days. In tensile strength, 0.64MPa (92 psi) and the maximum elongation were [640% and the Shore A durometer of this silicone emulsion] 22. Furthermore it aged for 18 hours, on the glass as a substrate, concrete, the pine that carried out the painting cloth, and a U.S. Japan cedar, other samples were formed in 50 mm in length, and width of 18 mm, and will be stiffened under a laboratory condition for 14 days. This elastomer showed the adhesive property (cohesive failure mode) excellent in all the substrates.

[Translation done.]